

(19)



Europäisches Patentamt  
European Patent Office  
Office européen des brevets

(11) Publication number:

**0 159 628**  
**A2**

(12)

## EUROPEAN PATENT APPLICATION

(21) Application number: 85104416.4

(51) Int. Cl.<sup>4</sup>: **A 61 K 7/11**

(22) Date of filing: 11.04.85

(30) Priority: 12.04.84 US 599296

(71) Applicant: Revlon, Inc., 767 Fifth Avenue, New York,  
N.Y.10022 (US)

(43) Date of publication of application: 30.10.85  
Bulletin 85/44

(72) Inventor: Stadnick, Richard P., 108 Derfuss Lane,  
Blauvelt New York (US)

(84) Designated Contracting States: AT BE CH DE FR GB IT  
LI LU NL SE

(74) Representative: Körber, Wolfhart, Dr. et al,  
Patentanwälte Dipl.-Ing. H. Mitscherlich Dipl.-Ing. K.  
Gunschmann Dr.rer.nat. W. Körber Dipl.Ing. J.  
Schmidt-Evers Dipl.-Ing. W. Melzer Steinsdorfstrasse 10,  
D-8000 München 22 (DE)

(54) Hair strengthening and permanent waving composition.

(57) Composition for strengthening hair and for imparting a  
permanent configuration thereto comprising an alkyltrialkoxysilane and a solvent.

**EP 0 159 628 A2**

ACTORUM AG

HAIR STRENGTHENING AND PERMANENT WAVING COMPOSITION

1 This invention relates to hair strengthening and permanent waving compositions, and more particularly, to reactive silicone hair strengthening and waving compositions.

5 Hair care is one of the most important parts of beauty care, and a large variety of hair treatment products are used for providing such care in the form of pre-shampoo conditioners, shampoos, after-shampoo conditioners, rinses, setting lotions, sprays, dyes, bleaches, permanent wave agents and the like. These products in addition to providing  
10 the desired result in the hair, such as luster, curl combability, softness, color and an overall appealing look, also do damage to the hair, especially when used indiscriminantly without professional guidance. Damage to hair may also result from other sources, such as combing,  
15 humidity, dryness, dirt, sunrays, such as u.v. and infrared radiation, and pollution in the atmosphere. However, damage to hair occurs mostly in the form of physical and chemical changes in hair as a result of bleaching, oxidative dying, hair relaxing via alkaline relaxers and reducing waving and  
20 curling preparations.

Hair damage may occur slowly and in various ways with frequent "treatment" of the hair. Over an extended time period the use of various chemicals in the hair treatment products will effect a weakened hair structure. Illustrative  
25 of this undesirable effect is that which occurs when the natural configuration of hair is altered from curled to straight or vice versa. The hair is subjected to the action of a composition which relaxes the hair by rupturing the disulfide bonds in the keratin of the hair to produce free  
30 sulfhydryl groups. This breakage in the disulfide diminishes

1 the rigidity of the hair proteins, leaving the hair pliable  
and soft. The compositions most commonly used as relaxers  
are based on sodium hydroxide, sulfite, or thioglycolate.  
When a thioglycolate based composition is used as the  
5 relaxer, the hair is treated with an oxidant, such as  
peroxide, to impart rigidity to the hair. These operations,  
particularly the reduction step, can be carried out at either  
room or elevated temperature. Both types of treatment, the  
cold as well as the hot, require visual evaluation of the  
10 hair. Ultimately, the quality of the hair, straightened or  
curled, rests on the subjective judgement of the salon  
operator, and more often than not because of the subjective  
element, the hair is either underprocessed or overprocessed.

In addition to the problem of being underprocessed  
or overprocessed, the hair subjected to oxidative hair  
15 preparations, lacks the desired texture, it is raspy,  
troublesome to comb, and require further conditioning. To  
correct these conditions some manufacturers of hair treatment  
products frequently supply post-treatment conditioners  
usually as part of a kit included with the acidic oxidative  
20 preparation. Others incorporate specific hair conditioning  
agents compatible with the oxidizing agents, such as  
non-ionic and cationic agents.

Hair conditioning agents are used not only in  
conjunction or after treatment with oxidizing agents but also  
25 to generally assist in the control and management of hair as  
for example after shampooing. Conditioned hair is easily  
untangled and combed through after shampooing, lays orderly  
when dry and provides a favorable feeling to the touch. The  
conditioning action on hair, particularly by cationic  
30 conditioning agents, is believed to be caused by the  
attraction of the positively charged agent to the negative  
sites on hair protein resulting in the deposition of the  
agent on to the hair fiber.

35

Cationic agents or surfactants have been used extensively as hair conditioning agents in creme rinses and shampoos. Best results have been obtained with cationic surfactants that are long chain high molecular weight quaternary compounds or long chain fatty amine salts. The positive charge of the quaternary surfactant is attracted to the negatively charged surface of the hair protein giving it lubricity during wet combing and a desirable texture after drying.

Other types of agents for deposition on hair used in the prior art are the organo silicones. The organo silicones, having hydrolyzable groups on the silicon atom, hydrolyze to the corresponding silanol and condense to an organo-silicone polymer on the hair fibers coating the same and providing body thereto are used for both conditioning and setting the hair.

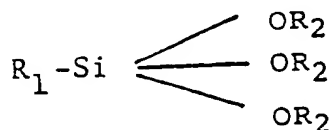
While the above mentioned conditioning agents provide substantial benefits to hair, none is directed to strengthening the hair structure and thereby restore the hair, as much as possible, to its natural strength.

The foregoing discussion illustrates some of the problems associated with hair care and solutions provided therefor by the prior art.

The present invention addresses the problem of treating the weakened hair, whether such condition is caused by nature, the environment or hair treatment preparations applied thereto.

The present invention relates to a composition for strengthening the tensile strength of hair comprising

a, an alkyltrialkoxysilane having the formula



wherein

$R_1$  is an aliphatic radical of  $C_1 - C_{18}$  carbon atoms, such as methyl, ethyl, propyl, butyl, hexyl, octyl, decyl, octadecyl; substituted aliphatic radicals selected from the group consisting of 3-aminopropyl, N,N-diethyl-3-aminopropyl, and N-(propyl) urea; and an alkenyl radical, such as vinyl and allyl;

$R_2$  is an aliphatic radical of  $C_1 - C_4$  carbon atoms, such as methyl, ethyl, propyl and butyl;

b, a solvent for said alkyltrialkoxysilane. The solvent may consist of water, ethanol, isopropyl alcohol, nonoxynol-4, or an aqueous solution of ethanol, isopropyl alcohol or nonoxynol-4.

To obtain a composition according to the present invention, a solution is prepared by adding an alkyltrialkoxysilane to the appropriate solvent with mixing. The composition preferably comprises about 0.5 to 40% v/v and more preferably 2 to 10% v/v of an alkyltrialkoxysilane in the solvent. The preferred solvent is an aqueous solution of ethanol, isopropyl alcohol or nonoxynol-4 in which the water constitutes no more than about 20% v/v of the total solvent volume.

Although the silanes described herein are stable for several days in alcohol/water phases having from 0 - 20% water, it is preferred to prepare the solution just prior to application to the hair. If the solution is allowed to stand in the presence of water for prolonged time periods, excessive polymerization and premature precipitation of the silane may occur, thus rendering the composition less effective for its intended use.

1 The method of application of the composition  
includes applying the composition to the hair by submerging  
the clean hair in the composition or applying the composition  
with an applicator and working it into the hair with the  
5 fingers and a comb. The process is completed by blow drying  
the hair with hot air.

The present invention is based on the discovery  
that the reactive alkyltrialkoxysilane containing three  
labile alkoxy groups in the presence of water hydrolyzes to  
10 form three reactive silanol groups which in turn condense  
with each other to form oligomers. The oligomers then  
hydrogen bond with functional groups containing active  
hydrogens (hydroxyl, amino, carboxyl, sulfhydryl and the  
like) in the hair. During drying or curing a stable covalent  
15 bond is formed with the hair with concomitant loss of water.  
While the curing process will occur at room temperature, it  
is preferred to speed up the reaction by drying the hair with  
a hot air dryer. The application of heat speeds up  
evaporation of the solvent and the by-products of the  
20 reaction. Once water has been removed from the hair, the  
resulting bonds become stable to hydrolytic action. The  
degree and rate of polymerization of the silane is determined  
by the amount of water available and the organic substituent  
of the silane.

25 This characteristic of the silanes of the present  
invention allows for their application to hair for increasing  
hair strength, retaining hair configuration such as curls,  
and providing manageability to hair.

The silanes of the present invention are either  
commercially available or can be prepared by methods known in  
30 the art using commercially available starting materials and  
reagents.

1 Silanes are readily solubilized in ethanol,  
isopropyl alcohol and many other organic solvents, however,  
for the present invention the preferred solvent for the  
silanes is an aqueous/alcohol mixture containing up to 20% by  
5 volume of water. This composition ensures that a sufficient  
amount of water is available to hydrolyze the silane and  
possibly swell the hair fibers for increased penetrations of  
the active ingredient prior to the polymerization step.  
Silanes show unexpected stability to water in such vehicles  
as determined by the time to hazing (precipitation of  
10 siloxane polymers). For example, solutions of  
ethyltriethoxysilane dissolved in 80/20 ethanol/water remain  
clear for several days. As the percentage by volume of water  
increases beyond 20% of the total solvent composition, more  
rapid hazing and precipitation of the siloxane polymer is  
15 observed to occur, thus rendering the composition less  
effective for its intended use.

Test methods used in connection with the invention  
and working examples follow.

#### Tensile Strength

20 This test relates to the determination of the force  
required to break (tensile strength at breakpoint) the hair  
sample by using a constant elongation rate tester such as the  
Instron Tensile Tester. The test is performed as follows:

25 Each tress of hair to be tested is conditioned in a  
50% relative humidity chamber for at least 24 hrs prior to  
use. Twenty strands of hair, randomly selected from each  
tress, are used for the test. A two-inch long section is cut  
from each hair strand and weighed to the nearest 0.1  
microgram. The weighed cut sections are placed into  
30 distilled water and allowed to hydrate for at least 16 hours.

35

1 Each hydrated hair sample is placed between the jaws of the Instron Tensile Tester, which are preset at 1 inch apart, and clamped securely. The tensile strength is then determined according to the manufacturer's instructions.

5 The tensile strength is the force required to break the hair strand. This force is expressed in terms of grams force per cross sectional area of hair strand.

Method For Determination of Silane Content in Hair

10 The silicon content of treated hair is determined colorimetrically as silicomolybdate or as molybdenum blue and the result calculated as silane. Approximately 0.7 gram  $\pm$  0.1 mg of hair is weighed accurately into a beaker, to which 5 ml of concentrated nitric acid is added. The sample is heated to dryness and the addition of nitric acid and heating steps are repeated until a white residue remains in the  
15 beaker. Then 10 ml of 1:10 hydrochloric acid is added to the beaker and the content is filtered with suction through a membrane filter. The beaker is thoroughly washed with 1:10 hydrochloric acid and the liquid is filtered. The filter is placed in the bottom of a polyethylene beaker and 1.0 ml of  
20 hydrofluoric acid is added to cover the filter. The beaker is then covered and allowed to stand for 30 min. Then 25 ml of water and 50 ml of boric acid solution is added with stirring, and the solution is heated at 40°C in a water bath for at least 10 min. Four (4) ml of molybdic acid reagent is  
25 added while stirring. Then 20 min. later, 20 ml of 10N sulfuric acid is added with stirring. If any yellow color persists, the solution is read within 2 min. after acidification in a spectrophotometer at 420 nm against distilled water. If a colorless solution results, the  
30 solution is allowed to stand for 2 to 5 min. and 1 ml of 1-amino-2-naphthol-4-sulfonic acid reagent is added, which is then mixed and read after 20 min. at 820 nm.



1 A calibration curve in the yellow silicomolybdate  
concentration range (420 nm) is made by diluting 0.5, 1, 2  
and 3 ml aliquots of the stock silicon (1mg/ml) standard to  
25 ml in polyethylene beakers and proceeding as hereinbefore  
described from and including the step of heating the solution  
5 for at least 10 min. After completing the steps as  
indicated, absorbance is read at 420 nm. Absorbance against  
mg silicon is then plotted. A calibration curve is also  
prepared in the molybdenum blue concentration range (820 nm)  
by diluting 50, 100, 200 and 500 microliters of the silicon  
10 stock standard (1mg/ml) to 25 ml in polyethylene beakers and  
proceeding as described above for the silicomolybdate  
calibration curve. Absorbance is read at 820 nm.

The concentration of silicon is obtained from the  
appropriate calibration curve. The calculation is as  
15 follows:

$$\text{Percent silane} = \frac{\text{mg silicon}}{\text{grams of hair}} \times \frac{\text{mole weight of silane}}{\text{atomic weight of silicon}} \times 0.1$$

20

25

30

35

EXAMPLE 1

1 A hair sample previously weakened by sodium  
hydroxide was submerged in 3.0% v/v solution of  
ethyltriethoxysilane (obtained from Petrarch Systems, Inc.,  
Levittown, PA) in a mixture of 80/20 ethanol/water. The hair  
5 was rinsed free of excess silane and cured by blow drying  
with a hot air dryer for about ten minutes. The hair was  
tested using the above-described test procedures. The  
results were as follows:

10 a. The hair treated with sodium hydroxide solution  
showed 11 to 14% decrease in tensile strength;

b. The sodium hydroxide weakened hair which  
subsequently was treated with the silane-containing  
composition increased in tensile strength back to within 97  
to 98% of its original (untreated) tensile strength;

15 c. The silane treated sodium hydroxide weakened  
hair picked up between 0.4 to 1.4% silicon by weight,  
calculated as ethyltriethoxysilane.

EXAMPLE 2

20 A 95% v/v solution of ethyltriethoxysilane in  
nonoxynol-4 was prepared. Immediately prior to treating the  
hair, 40 parts of the silane/nonoxynol-4 phase was added with  
stirring to 60 parts of water. The hair, which was  
previously weakened by sodium hydroxide was completely wetted  
with the silane/nonoxynol-4/water solution. The solution was  
25 allowed to remain on the hair for 30 minutes. The hair was  
rinsed free of excess solution and blow dried with a hot air  
drier.

30 Upon testing, the data showed a 10% increase in  
tensile strength of the hair.

EXAMPLE 3

1 A swatch of virgin hair was heavily damaged by a 45  
 minute treatment with a 2% aqueous solution of sodium  
 hydroxide. The alkali-damaged swatch was washed with a pH  
 7.0 shampoo to restore the acid balance, allowed to dry under  
 5 ambient conditions and subdivided into a series of  
 equal-sized tresses. Three tresses were selected and each  
 treated for 30 minutes with 10 grams of the silane-containing  
 composition of Example 2, cured and blow dried. The  
 10 procedure used to treat the three tresses was exactly the  
 same except for the manner in which the excess silane was  
 removed. Instron analyses were carried out on all of the  
 tresses. The result and the manner of removing excess silane  
 are shown in Table I.

15 Table I  
Instron Results for Prepared Tresses

|    | <u>Sample</u>   | <u>n</u> | <u>Tensile Strength (G)</u>   |              |
|----|---|----------|-------------------------------|--------------|
|    |   |          | <u><math>\bar{X}^*</math></u> | <u>SEM**</u> |
| 20 | Control virgin hair   | 20       | 29.49                         | $\pm 0.61$   |
|    | Control alkali-damaged hair   | 20       | 14.48                         | $\pm 1.07$   |
|    | Alkali-damaged hair,<br>silane treated, rinsed,<br>then cured                 | 20       | 17.51                         | $\pm 0.56$   |
| 25 | Alkali-damaged hair,<br>silane treated, cured,<br>then rinsed                 | 20       | 16.56                         | $\pm 0.59$   |
| 30 | Alkali-damaged hair<br>silane treated, cured,<br>then rinsed and<br>shampooed | 20       | 16.95                         | $\pm 0.702$  |

n denotes the number of determinations here and in subsequent tables.

$\bar{X}^*$  denotes the mean of the number of measurements here and in subsequent tables.

35 SEM\*\* denotes the standard error of the mean here and in subsequent tables.

1 The data in Table I show that all the silane  
treated tresses have significantly increased in strength  
(about 15-20%) relative to the alkali-relaxed control. No  
significant difference in hair strength is shown among the  
silane-treated tresses indicating that the mode by which  
5 excess reagent is removed from the hair has no affect on hair  
strength.

It was also discovered that if hair, arranged in a  
desired configuration with curling rollers or rods, is  
treated with a silane-containing composition of the present  
10 invention and blow dried, the silane bonding to the hair not  
only increases hair strength but also maintains the hair in  
the shape in which it was dried. This effect is shown for  
both alkali-weakened and virgin hair. The resulting  
permanent wave resists the tendency to revert to the initial  
15 configuration even after repeated shampooing.

EXAMPLE 4

A dark brown virgin hair swatch obtained from a  
single lot was washed with a 10% sodium lauryl sulfate  
solution, rinsed with tap water, and blow dried at room  
20 temperature. The swatch was subdivided into eighteen tresses  
ten inches long each weighing one gram. One tress was  
selected to serve as virgin control and five other were set  
aside for silane treatment. The remaining twelve tresses  
were set aside for the alkali treatment. Before treatment,  
25 each tress was gently combed and separately rolled on curling  
rods. Alkali treatment was carried out by submerging the  
rolled tresses in a 2.0% sodium hydroxide solution for one  
hour. Subsequently, each tress was rinsed under running tap  
water for at least 10 minutes. The rolled tresses were towel  
30 dried, soaked in pH 7.0 buffer for one hour to restore the  
acid balance, and rinsed out thoroughly to remove the buffer.  
The rolled tresses were finally dried at room temperature.

Equimolar solutions of silanes denoted in Tables II  
1 and III were prepared separately in a mixture of 80:20  
ethanol/H<sub>2</sub>O, to carry out silylation. The virgin and alkali  
treated tresses on rollers were treated separately for 30  
5 minutes with 100 ml of 0.26M solution of silane. The rolled  
tresses were blow dried for four minutes after the silane  
treatment with a 1600-watt blow drier and thoroughly sprayed  
with water. The rolled tresses were allowed to stand for  
five minutes and finally blow dried for 15 minutes at the  
10 high temperature setting. Unsilylated virgin and alkali  
control were also subjected to the same treatment. The hair  
tresses, after equilibrating to room temperature, were  
carefully unwound from the curling rods and mounted on the  
curl retention board. The mounted specimens were then  
15 incubated at 100°F/95% relative humidity. Curl retention  
data were obtained periodically over 24 hours. The tresses  
were removed from the board, wetted under running tap water  
for 1 minute, dried and soaked in a beaker of water for 30  
additional minutes, removed, air dried, twice shampooed and  
rinsed again. The final curl length and patterns of each  
20 tress were evaluated to determine curl stability. Data and  
comments are summarized in Tables II and III.

25

30

35

Table II  
Alkali Pre-Treated Hair

| Sample                           | Silane (1)<br>Conc.<br>(%) | n | % Curl<br>Retention<br>(X) | SEM   | Comments                                 |
|----------------------------------|----------------------------|---|----------------------------|-------|--|
| Control (alkali<br>relaxed)      | 0.00                       | 2 | 12.27                      | +1.35 | Good curl and S-wave pattern<br>No shine |
| Octadecyl<br>triethoxysilane     | 10.84                      | 2 | 14.55                      | +0.13 | Very good S-wave pattern<br>Good shine   |
| 3-Aminopropyl<br>triethoxysilane | 4.70                       | 2 | 17.80                      | +0.20 | Very good S-wave pattern<br>Good shine   |
| N-(triethoxy<br>silylpropyl)urea | 6.90                       | 2 | 16.36                      | +0.42 | Very good curl and S-wave<br>pattern     |

(1) All solutions contain equal moles of silanes (0.26M)

Table - III

Virgin Hair

| <u>Sample</u>                | <u>Silane (1)<br/>Conc.<br/>(%)</u> | <u>n</u> | <u>% Curl<br/>Retention<br/>(x)</u> | <u>Comments</u>                   |
|------------------------------|-------------------------------------|----------|-------------------------------------|-----------------------------------|
| Control virgin hair          | 0.00                                | 1        | 7.16                                | Almost no curl, no S-wave pattern |
| Octadecyltriethoxy-silane    | 10.84                               | 1        | 12.73                               | Very good body wave               |
| Octyltriethoxy-silane        | 7.20                                | 1        | 11.04                               | Fair body wave                    |
| 3-Aminopropyltriethoxysilane | 4.70                                | 1        | 11.35                               | Good body wave                    |
| Vinyltriethoxy-silane        | 4.90                                | 1        | 11.94                               | Good body wave                    |
| N-(triethoxysilylpropyl)urea | 6.90                                | 1        | 10.38                               | Fair body wave                    |

(1) All solutions contain equal moles of silanes (0.26M)

EXAMPLE 5

1           Series of tresses were prepared from a single lot  
of dark brown virgin hair, each tress being  $1.5 \pm 0.1g$  in  
weight and 10" long. Two of the tresses, selected at random  
to serve as permanent waved controls, were treated with  
5 Collagen Amino Acid Professional Formula <sup>®</sup> (active  
ingredients: 9.2% ammonium thioglycolate, 10% sodium  
bromate). Fourteen virgin hair tresses were wrapped on rods  
and immersed for 1 hour in a 2% sodium hydroxide solution.  
The wrapped tresses, after having been rinsed free of alkali,  
10 were treated with pH 7.0 buffer to restore the acid balance  
to the hair. The tresses were rinsed free of buffer and  
treated as described in Tables IV and V. The procedure used  
for the evaluation and curing of alkali-treated hair was  
described in Example 4. Heat was also applied to the control  
15 samples for an identical period of time. All the prepared  
tresses were allowed to equilibrate under ambient conditions  
of temperature and humidity.

After equilibration, the tresses were carefully  
unwound from their curling rods and their respective curl  
20 lengths carefully measured to the nearest 0.1 cm. The  
tresses, after having been mounted on a curl retention panel,  
were incubated for 24 hours at 100°F/95% RH. Next, the  
tresses were removed from the panel, flushed with running tap  
water for 1 minute, soaked in a beaker of water for 30  
25 additional minutes, removed and shampooed twice the Flex  
Shampoo <sup>®</sup>, rinsed free of soap and allowed to air dry. The  
final curl length and pattern of each tress were carefully  
evaluated to determine curl stability.

Results are shown in Tables IV and V.



0159628

Table - IV  
Curl Retention

| Sample   | n | % Curl Retention (X) | SEM   | Comments                        |
|--|---|----------------------|-------|---------------------------------|
| Alkali relaxed control                                   | 1 | 10.2                 | -     | Little curl - no S-wave pattern |
| Alkali relaxed/5% ETES <sup>1</sup>                      | 1 | 15.4                 | -     | Good S-wave pattern             |
| Alkali relaxed/30% ETES <sup>1</sup>                     | 1 | 15.0                 | -     | Good S-wave pattern             |
| Alkali relaxed/urea swelled-30% ETES <sup>1</sup>        | 1 | 13.4                 | -     | Good S-wave pattern             |
| Alkali relaxed/salt swelled-30% ETES <sup>1</sup>        | 1 | 14.2                 | -     | Good S-wave pattern             |
| Alkali relaxed/30% ETES <sup>1</sup> in DMF <sup>2</sup> | 2 | 15.2                 | +0.20 | Very good S-wave pattern        |
| Alkali relaxed/30% ETES <sup>1</sup> in DMF <sup>3</sup> | 2 | 14.1                 | +0.20 | Very tight curl                 |
| Permanent waved controls                                 | 2 | 16.7                 | +0.60 | Very good S-wave pattern        |

(1) ETES = Ethyltriethoxysilane in 80/20 alcohol/water in here and in Table V

(2) DET = Diethyltoluamide

(3) DMF = Dimethylformamide

0159628

Table - V

| <u>Sample</u>                                 | <u>n</u> | <u>% Curl Retention (x)</u> | <u>SEM</u> | <u>Comments</u>                     |
|---|----------|-----------------------------|------------|-------------------------------------|
| Virgin hair control                           | 2        | 6.9                         | +0.60      | Very little curl, no S-wave pattern |
| Virgin hair/5% ETES                           | 2        | 12.2                        | +0.80      | Good body wave                      |
| Virgin hair/salt swelled 5% ETES <sub>1</sub> | 2        | 12.6                        | +0         | Good body wave                      |
| Virgin hair/urea swelled 5% ETES <sub>2</sub> | 2        | 12.6                        | +0         | Good body wave                      |
| Alkali relaxed control                        | 2        | 7.5                         | +0         | Very little curl, no S-wave pattern |
| Alkali relaxed/5% ETES                        | 2        | 12.2                        | +0         | Good S-wave pattern                 |
| Alkali relaxed/urea swelled 5% ETES           | 2        | 11.4                        | +0         | Good S-wave pattern                 |
| Alkali relaxed/salt swelled 5% ETES           | 2        | 10.4                        | +1.00      | Fair S-wave pattern                 |

- (1) Hair was swelled with a 2.8M LiCl/0.5M Na<sub>2</sub>SO<sub>4</sub> aqueous solution prior to treating the hair with silane.
- (2) Hair was swelled with a 4M aqueous solution of urea prior to treating the hair with silane.

1           Table IV demonstrates that silylation of alkali  
treated hair on curling rods produces a curl pattern  
equivalent to that produced by a conventional permanent  
waving treatment, and is substantially greater than the curl  
5           produced in the alkaline relaxed control. Curl "memory" was  
retained in the alkali-silylated tresses even after repeated  
shampooing.

          Table V demonstrates that silane compositions of  
the present invention impart a good stable curl to alkali  
treated hair and, in addition, that the curl retention of the  
10          virgin hair is significantly improved after silylation  
through the formation of a stable body wave pattern.

          It will be understood that modifications and  
variations may be made without departing from the spirit and  
scope of the novel concept of the present invention.  
15

20

25

30

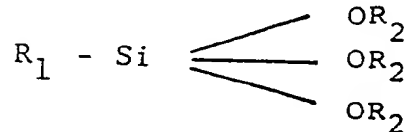
35

WHAT IS CLAIMED IS:

1 1. A composition for strengthening the tensile strength of hair comprising:

a., an alkyltrialkoxysilane having the formula

5



wherein

10  $R_1$  is an aliphatic radical of  $C_1 - C_{18}$  carbon atoms; substituted aliphatic radicals selected from the group consisting of 3-aminopropyl, N,N-diethyl-3-aminopropyl, N-(propyl)urea and alkenyl;

15  $R_2$  is an aliphatic radical of  $C_1 - C_4$  carbon atoms; and

b., a solvent for said alkyltrialkoxysilane.

2. The composition of claim 1 wherein the aliphatic radical is methyl, ethyl, propyl, butyl, hexyl, octyl, decyl or octadecyl.

20 3. The composition of claim 1 or 2 wherein the alkenyl is vinyl or allyl.

4. The composition of any of claims 1 to 3 wherein the solvent is water, ethanol, isopropyl alcohol or nonoxynol-4.

25 5. The composition of any of claims 1 to 3 wherein the solvent is an aqueous solution containing ethanol, isopropyl alcohol or nonoxynol-4.

6. The composition of clam 5 wherein water constitutes no more than about 20% v/v of the solvent.

30 7. The composition of any of claims 1 to 6 wherein the alkyltrialkoxysilane is present in about 0.5 to 40% v/v based on the total volume of the composition.

35

1           8. The composition of any of claims 1 to 7 wherein  
based on the total volume of the composition  
alkyltrialkoxysilane is present in an amount from about 2 to  
10% v/v in 98-90% v/v of a solvent.

5           9. The composition of any of claims 1 to 8 wherein  
the alkyltrialkoxysilane is octadecyltriethoxysilane,  
n-octyltriethoxysilane, 3-aminopropyltriethoxysilane,  
vinyltriethoxysilane, n-(triethoxysilylpropyl) urea or  
ethyltriethoxysilane.

10          10. A method of strengthening hair comprising the  
steps of:

a., applying to the hair an effective amount of the  
composition of any of claims 1 to 9

b., applying heat to the hair to effect curing.

15          11. The method of claim 10 wherein the hair is an  
alkali-weakened hair.

12. The method of claim 10 or 11 wherein the hair  
is virgin hair.

20          13. The method of any of claims 10 to 12 wherein  
prior to step (a) the hair is set to the desired  
configuration.

14. The method of any of claims 10 to 13 wherein  
the hair is pre-treated with an alkali or a thioglycolate  
prior to applying the composition.

25

30

35

**THIS PAGE BLANK (USPTO)**